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12/18/02 jc806 U.S. PTO

JC996 U.S. PTO 60/433970

PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR 1.53 (c).

Express Mail Label No.

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☐ *Additional inventors are being named on the _____ separately numbered sheets attached hereto*

TITLE OF THE INVENTION (280 characters max)

SMALL VOLUME VIAL

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ENCLOSED APPLICATION PARTS *(check all that apply)*

☒ Specification *Number of Pages* 9 ☐ CD(s), Number

☒ Drawing(s) *Number of Sheets* 2 ☐ Other (specify)

☐ Application Data Sheet. See 37 CFR 1.76

METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT *(check one)*

☒ Applicant claims small entity status. See 37 CFR 1.27.

☒ A check or money order is enclosed to cover the filing fees

☐ The Commissioner is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number:

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FILING FEE AMOUNT ($): 80.00

The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.

☒ No.

☐ Yes, the name of the U.S. Government agency and the Government contract number are: _____.

Respectfully submitted,

SIGNATURE [signature]

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Date 12/18/02

REGISTRATION NO. *(if appropriate)* 35,094

Docket Number: 12090-000011/US

USE ONLY FOR FILING A PROVISIONAL APPLICATION FOR PATENT

TITLE

Small Volume Vial

FIELD OF THE INVENTION

The present invention relates to a small volume vial, and more specifically to a sealed vessel assembly for performing microwave-assisted chemical reactions on small reaction mixture volumes. The invention also refers to a system for performing microwave-assisted chemical reactions on small reaction mixture volumes contained in the sealed vessel assembly, and to the use of the sealed vessel assembly and system for performing microwave-assisted organic synthesis reactions.

BACKGROUND AND PRIOR ART

The expression "microwave-assisted chemical reactions" as used herein refers to processes utilizing microwaves to initiate or accelerate chemical reactions. Microwave-assisted chemistry is used for heating materials in a variety of chemical processes, the microwaves interacting directly with the materials including water and a number of organic liquids to cause molecular action and generate heat. Accordingly, microwave-assisted techniques have been developed both for analytic and synthetic processes. The equipment used for performing microwave-assisted chemistry conventionally includes an apparatus having a cavity into which microwaves are guided from a microwave source, typically a magnetron. A vessel, containing the reactants, is introduced in the cavity and positioned for exposure to the microwaves. In some appliances, the vessel is a sealed container in which a reaction can proceed under conditions of both elevated temperature and elevated pressure. The vessel, and the apparatus for exposing the reaction mixture in the vessel to microwaves, provide the basic components in a system for performing microwave-assisted chemical reactions. In commercial practice, the system additionally comprises means for vessel transport to and from the cavity, means for dispensing solvent and reactants to the vessel, detection means, power and control means, and a rack for parking the vessel outside the cavity.

A commercially available reaction vessel in a system for performing microwave-assisted chemical reactions is diagrammatically shown in Figure 1 of the drawings,

attached hereto. This known reaction vessel comprises the well known shape of a test tube, having a penetrable septum capped over the open end of the tube. The tube diameter is dimensioned to fit with other components of the system such as the cavity entrance, gripper means and a parking rack . The vessel has a reaction chamber dimensioned for containment of reaction mixture volumes commonly processed in the system.

In this context, the expression "small reaction mixture volumes" refers to reaction mixture volumes in a micro liter range, such as volumes down to 500 μl or less, for example 1-500 μl, preferably 100-500 μl. Specific problems need to be addressed when designing a reaction vessel for performing microwave-assisted chemistry on small volumes contained in the vessel. For example, the geometry in a top portion of the vessel should be designed to facilitate reflux/re-flow of fluid; the inner diameter of the vessel should be large enough to avoid formation of meniscus of fluid that would be unable to flow back from the top of the vessel; the container volume that is not occupied by the reaction mixture, or head space, should be related to the volume of reactants to avoid exaggerated vaporization at operative pressures and temperatures; in spite of reduced containment dimensions, the vessel exterior must match the cavity entrance diameter and the transporting gripper means, as well as parking racks, and be able to position the reaction mixture for dispensing of reactants and/or solvents, microwave exposure, and detection.

The present invention aims to solve these and other problems encountered when performing microwave-assisted chemical reactions on small volumes.

A first object of the invention is therefore to provide a reaction vessel that allows microwave-assisted chemistry to be performed on small volumes.

Another object is to provide a system that allows microwave-assisted chemistry to be performed on small volumes by incorporating the reaction vessel of the invention.

These and other objects and aims are met in a vessel assembly and a system according to the appended claims.

SUMMARY OF THE INVENTION

Briefly, the present invention suggests a vessel assembly for performing microwave-assisted chemical reactions on small volumes, wherein a reaction vessel is sealed through a penetrable and self-sealing diaphragm that is capped over an open end of the reaction vessel. The reaction vessel mouths in an end plane of a sleeve surrounding the reaction vessel, the diaphragm being clamped to the end plane through a ring shaped cap engaging an outer boundary of the sleeve. The sleeve provides a radial extension of the reaction vessel in order to bridge the radial distance between a wall of the reaction vessel and other components in a system for performing microwave-assisted chemical reactions.

BRIEF DESCRIPTION OF THE DRAWINGS

The vessel assembly and system are further described below with reference to the attached drawings. In the drawings,

Fig. 1 is a diagrammatic presentation of a system for performing microwave-assisted chemical reactions, and a prior art reaction vessel adapted to the system;

Fig. 2 shows an embodiment of the new vessel assembly in an exploded perspective view;

Fig. 3 is a longitudinal section through the sealed vessel assembly according to a preferred embodiment of the present invention, and

Fig. 4 is a sectional view similar to that of Fig. 3, showing a modified embodiment of the sealed vessel assembly.

DETAILED DESCRIPTION OF THE DRAWINGS

A preferred embodiment of the present invention will be more fully described below, modifications thereto successively outlined as the disclosure proceeds.

With reference to Fig 1, a system for performing microwave-assisted chemical reactions typically comprises an apparatus formed with a cavity 1, here shown with a prior art reaction vessel 2 being inserted and vertically positioned for exposure to microwaves from a microwave source (not shown). In the inserted position, the reac-

tion vessel 2 penetrates the effective measurement area of a detecting means, such as an infrared light (IR) sensor 3. The reaction vessel 2 has an outer diameter adapted to a diameter D of an entrance to the cavity. The entrance is here represented by an insertion guide 4, determining the diameter D through its inner radius. Though not shown in the drawing, a gripper means for vessel transport to and from the cavity, as well as a rack for parking the vessel outside the cavity are likewise engineered to operate with the diameter D.

Fig. 2 illustrates an embodiment of the new vessel assembly for performing microwave-assisted chemical reactions on small volumes in a system substantially as mentioned above. The vessel assembly according to the invention comprises a reaction vessel 10, a sleeve member 20, a penetrable diaphragm 30 and a cap member 40 as its main components.

The reaction vessel 10 has an open upper end and a closed bottom end, and an inner volume 11 (Figs. 2, 3) determined in respect of the small volumes to be contained therein for chemical reactions initiated or accelerated by microwaves. In most cases the reaction vessel 10 preferably is a circular cylinder, however, other cylinders having rounded or polygonal sections may likewise be applied to take advantage of the invention disclosed herein. Accordingly, an inner diameter d of reaction vessel 10 is dimensioned to permit effective microwave absorption by the reactants and solvents in the vessel, and to avoid formation of meniscus of fluid that would stick to the wall of reaction vessel 10 and be hindered to flow back from the top of the vessel. As a non-limiting example, the reaction vessel 10 may have an inner diameter d in the range of a few millimeters, such as 3-5 mm, or as preferred not less than 3,5 mm.

The volume that is not occupied by solvent and reactants contained in the reaction vessel 10, i.e. the headspace volume of reaction vessel 10, is dimensioned in relation to the smallest reaction mixture volume to be handled in order to avoid exaggerated vaporization at operative pressures and temperatures. As a guidance, the head space volume preferably should be less than 20 times the smallest volume processed in the reaction vessel 10. The bottom of reaction vessel 10 may have any preferred shape such as flat, conical, rounded, etc., and formed in the terminal end 13 of the reaction vessel as illustrated in Fig. 4 of the drawings. Alternatively, the axial length l of reaction vessel 10 may be determined through a radial compression

12, forming a closed bottom above the terminal end 13 of the reaction vessel as illustrated in Figs. 2 and 3, respectively.

The geometry in the upper end of reaction vessel 10 is determined in order to facilitate reflux of liquid and to minimize the risk of solvent and/or reaction mixture sticking to the inner surface of the reaction vessel. Most preferred, the top portion of reaction vessel 10 is formed with increasing radius towards the open end, forming a conical mouth 14. For example, the conical mouth 14 may alternatively have a progressively increasing radius forming a flared cone (not shown) in the top portion of reaction vessel 10. In all alternatives, the inner wall of reaction vessel 10 connects to the mouth of increased radius through a continuous transition region 15. The mouth 14 has a flange or rim 16, defining the opening in the upper end of reaction vessel 10 and contemplated for a sealed engagement with the penetrable diaphragm 30 as discussed below.

The sleeve member 20 is a cylinder with an outer perimeter corresponding to the diameter D, running from an upper end plane 21 to a bottom end 22, the bottom end being chamfered or formed with a radius 23. A seat 24 is formed in the end plane and correspondingly shaped to receive and support the top portion of increased radius of reaction vessel 10. In the received position, the reaction vessel 10 extends centrally through the sleeve 20, the sleeve providing a radial extension of the reaction vessel in concentric relation about the vessel to bridge the radial distance between the wall of reaction vessel 10 and the operative diameter D of the system. Optionally, the seat 24 and reaction vessel 10 may be offset from the center of end plane 24. The axial length of sleeve 20 is determined with respect to conditions set by the system, such as the structure of parking racks, gripper means, and microwave cavity specifications. Thus, the sleeve may be of same axial length as the reaction vessel 10, or extend beyond the terminal end of the reaction vessel, or extend only for a part of the axial length of the reaction vessel 10.

The upper end of sleeve 20 is formed with a groove or other formation, such as a flange 25, for holding the cap member 40 over the end plane 21. The cap 40 is a ring shaped element having a through hole 41 positioned to match the mouth 14 of reaction vessel 10 in the assembled position. The cap advantageously is a metal element that is crimped about the outer boundary of the end plane 21, in the illustrated embodiment for a firm engagement with the lower side of the flange 25.

The penetrable diaphragm 30 is clamped between the cap 40 and end plane 21 of the sleeve 20 in the sealed vessel assembly. In the clamped position, the diaphragm 30 covers the mouth 14 of reaction vessel 10 and extends in radial direction outwardly of the rim 16. The diaphragm 30 preferably has a radius corresponding to that of the end plane 21. The rim 16, protruding slightly above the end plane 21 when the reaction vessel 10 is accommodated in the sleeve 20 as illustrated in Fig. 3, is thus depressed in the lower side of the diaphragm for a sealed enclosure of the inner volume 11 of reaction vessel 10. The diaphragm 30 is an elastic element having self-sealing properties after penetration from a dispensing means, such as a needle. Suitable materials for the diaphragm 30 are well known within the art and need not be further commented here.

Reaction vessel 10 is microwave transparent and made for example of glass, and preferably also the sleeve 20 is made from microwave transparent materials such as glass or any suitable polymer material, such as a polyethylene-based polymer material. The sleeve 20 may have a homogenous section (Fig. 3) from the outer perimeter to the wall of reaction vessel 10 or may alternatively be formed with a thin wall section (Fig. 4), depending from the end plane 21 and enclosing an annular cavity radially about the wall of the reaction vessel 10. In the presently preferred embodiment, the reaction vessel 10 and sleeve 20 are separate elements fixedly secured through the cap 40 and diaphragm 30, the diaphragm being clamped by the cap to engage the rim 16 and firmly position the increased diameter portion of the reaction vessel in the seat of the sleeve. Alternatively, the reaction vessel and sleeve may be integrally formed from one material as illustrated in Fig. 4. The appended claims are drafted to include any and all of such modifications that would appear obvious to the man skilled in the art after reading this specification.

CLAIMS (2002-12-12)

1. A sealed vessel assembly for performing microwave-assisted chemical reactions on small volumes, the assembly comprising a reaction vessel (10) having an open upper end and a closed bottom end, a microwave transparent sleeve (20) in concentric relation about the reaction vessel, the sleeve having an outer perimeter and an end plane (21), the outer perimeter of the sleeve defining a radial extension of the reaction vessel, a ring shaped cap (40) secured over the end plane of the sleeve, and a penetrable diaphragm (30) clamped between the cap and the sleeve for sealing the open end of the reaction vessel, mouthing in the end plane of the sleeve.

2. The assembly of claim 1, wherein an axial length of the sleeve (20) is less than the total length of the reaction vessel (10).

3. The assembly of claim 1 or 2, wherein the sleeve (20) and reaction vessel (10) are separate elements fixedly secured by the ring shaped cap (40) and the diaphragm (30).

4. The assembly of claim 3, wherein the upper end of the reaction vessel has a portion of increased diameter (14,15,16), and the end plane of the sleeve is formed with a corresponding seat (24) for the increased diameter portion of the reaction vessel.

5. The assembly of claim 4, wherein the seat in the sleeve is dimensioned to accommodate the reaction vessel such that a rim portion (16), defining the opening in the upper end of the reaction vessel, protrudes above the end plane (21) of the sleeve to be impressed in the penetrable diaphragm (30) that is clamped to the sleeve for sealing engagement with the reaction vessel (10).

6. The assembly of claim 5, wherein the increased diameter portion of the reaction vessel is a flange.

7. The assembly of claim 5, wherein the increased diameter portion of the reaction vessel is a cone.

8. The assembly of claim 5, wherein the increased diameter portion of the reaction vessel is a flared cone.

9. The assembly of any previous claim, wherein the sleeve has an inner diameter defining an annular cavity about the reaction vessel.

10. The assembly of any previous claim, wherein a bottom of the reaction vessel (10) is formed through a radial compression (12) of the reaction vessel diameter above the terminal end (13) of the reaction vessel.

11. The assembly of any previous claim, wherein the reaction vessel has an inner volume including a head space volume which is less than 20 times that of the smallest reaction mixture volume contained in the vessel.

12. The assembly of any previous claim, wherein the reaction vessel is dimensioned for performing microwave-assisted chemical reactions on small volumes of 500 μl or less.

13. A system for performing microwave-assisted chemical reactions on small volumes, comprising a sealed vessel assembly (10,20,30,40) according to any previous claim 1-12.

14. The system of claim 13, wherein the outer perimeter of the sleeve is dimensioned for bridging the radial distance between a wall of the reaction vessel and an entrance diameter (D) of a microwave cavity (1) in the system.

15. The use of the assembly according to any of claims 1-12 for performing microwave-assisted chemical reactions, in particular microwave-assisted organic synthesis reactions.

17. The use of the system according to any of claims 13-14 for performing microwave-assisted chemical reactions, in particular microwave-assisted organic synthesis reactions.

- - -

ABSTRACT

A vessel assembly for performing microwave-assisted chemical reactions on small reaction mixture volumes is disclosed, wherein a reaction vessel (10) is sealed through a penetrable diaphragm (30) that is capped over an open end of the reaction vessel. The reaction vessel mouths in an end plane of a sleeve (20) surrounding the reaction vessel, the diaphragm being clamped to the end plane through a ring shaped cap (40) engaging an outer boundary of the sleeve. The sleeve provides a radial extension of the reaction vessel in order to bridge the radial distance between a wall of the reaction vessel and other components in a system for performing microwave-assisted chemical reactions.

Fig. 3

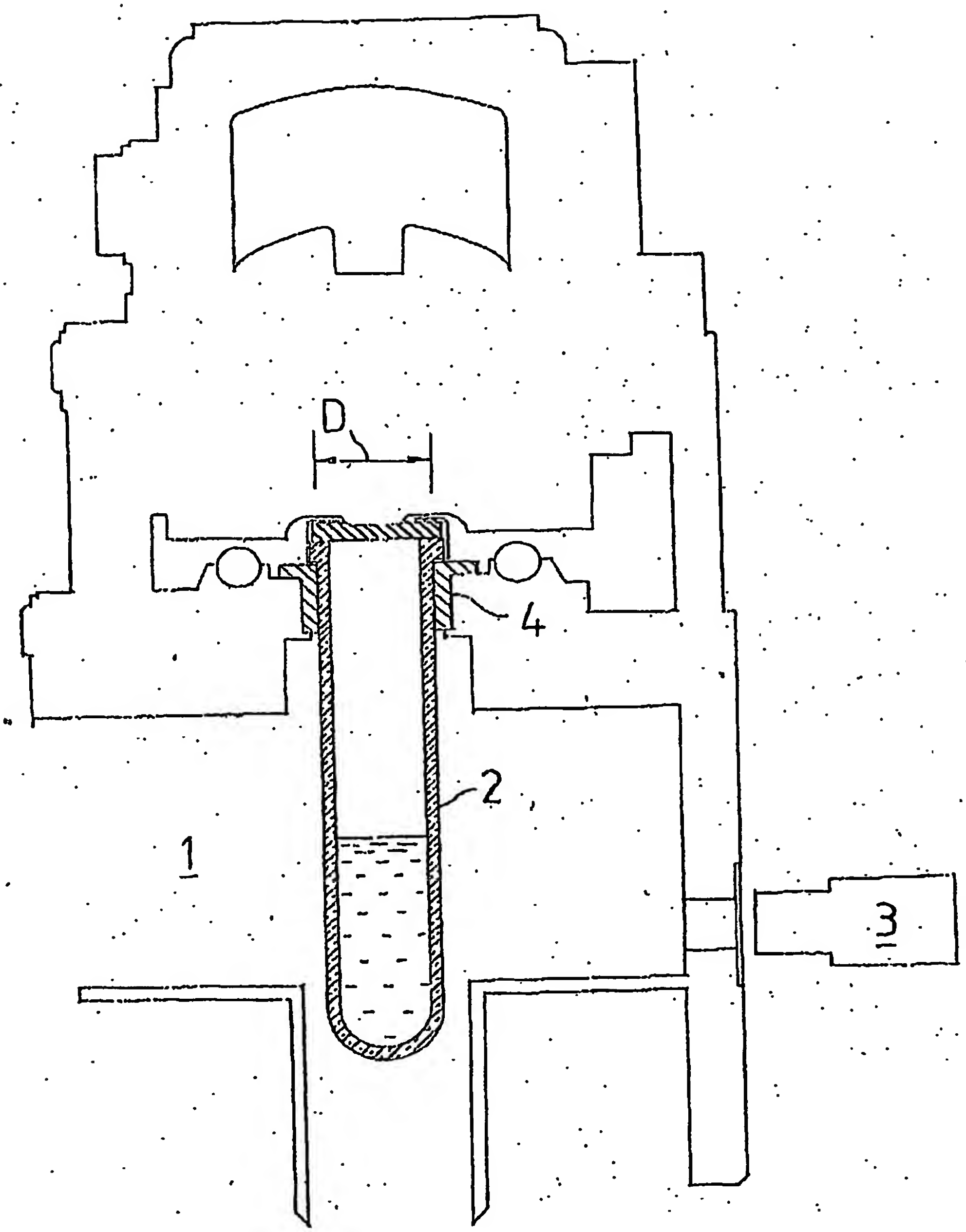

Fig.1 (Prior art vessel)

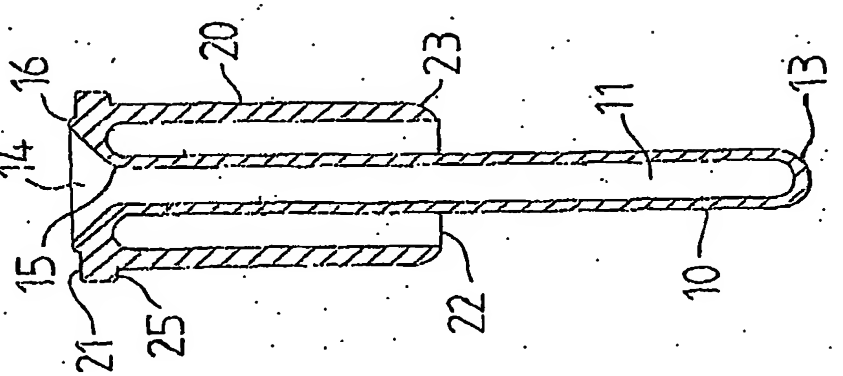

Fig. 4

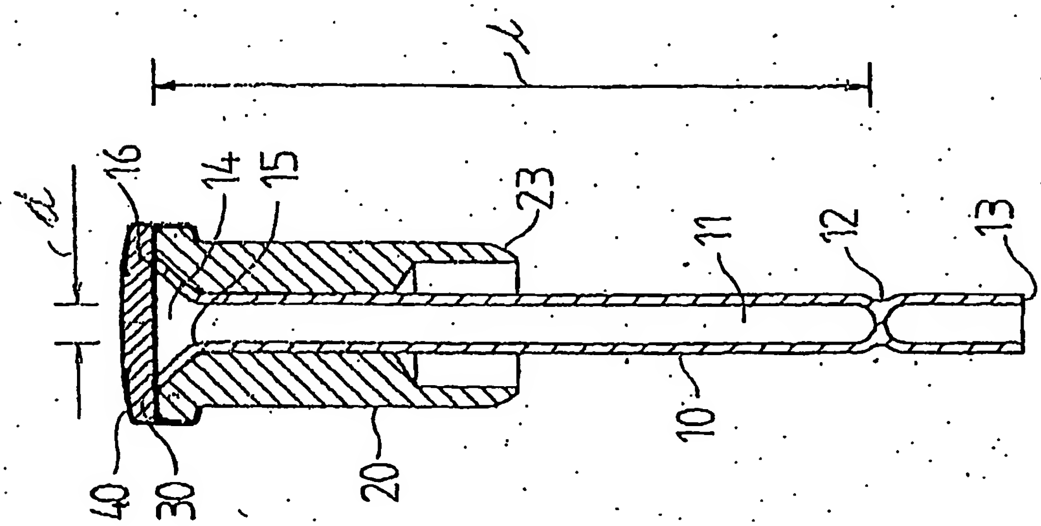

Fig. 3

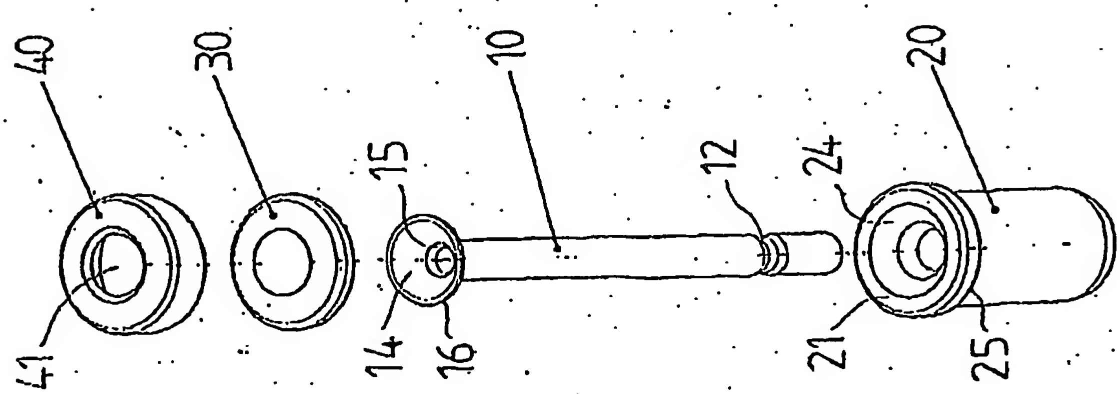

Fig. 2